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Key indicators

Single-crystal X-ray study
T = 291 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.052
wR factor = 0.076
Data-to-parameter ratio = 22.9

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Ethyl 5-chloro-9-ethoxy-2-oxo-2H-pyrano-[2,3-g]quinoline-8-carboxylate

The title compound, $\text{C}_{17}\text{H}_{14}\text{ClNO}_5$ has been shown to be isostructural with the bromo analogue, confirming the site of alkylation within the structure and the pyranoquinoline ring system. As with the bromo analogue, the molecules are linked by a $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond forming $C(5)$ chains along [001].

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Comment

Following from the successful bromination of a pyranoquinoline derivative (da Matta *et al.*, 2000; de Oliveira, 2003) at the carbonyl site of compound (1) (see scheme) (de Oliveira, 2006), chlorination was also attempted. As with the bromo compound (de Oliveira *et al.*, 2006), alkylation occurs at the carbonyl site (Fig. 1).



The structure also reveals the same hydrogen bonding scheme with intramolecular hydrogen bonds (Table 1) supporting the structure, and $C(5)$ chains (Bernstein *et al.*, 1995) forming along [001] (de Oliveira *et al.*, 2006).

Experimental

The title compound was obtained from the reaction between EtBr and (1) in DMF solution in the presence of K_2CO_3 (de Oliveira,

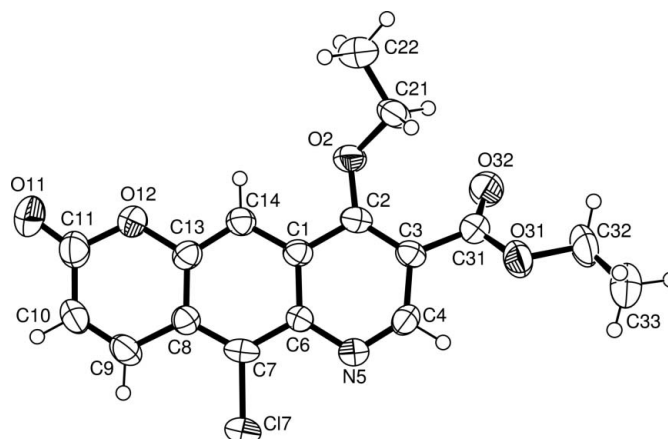


Figure 1

The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

2003). Pure product (2) was obtained from the reaction mixture by column chromatography using hexane–ethyl acetate as the eluent (gradient 1:4 to 1:1). Crystals suitable for X-ray crystallography were grown from ethyl acetate (65% yield; m.p. 433–434 K).

Crystal data

$C_{17}H_{14}ClNO_5$	Mo $K\alpha$ radiation
$M_r = 347.74$	Cell parameters from 1346 reflections
Orthorhombic, $Pna2_1$	$\theta = 3.5\text{--}19.3^\circ$
$a = 7.1480$ (9) Å	$\mu = 0.27$ mm $^{-1}$
$b = 19.653$ (3) Å	$T = 291$ (2) K
$c = 11.1150$ (14) Å	Needle, colourless
$V = 1561.4$ (3) Å 3	$0.38 \times 0.10 \times 0.02$ mm
$Z = 4$	
$D_x = 1.479$ Mg m $^{-3}$	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	5017 independent reflections
ω scans	1745 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$R_{\text{int}} = 0.100$
$T_{\text{min}} = 0.866$, $T_{\text{max}} = 0.995$	$\theta_{\text{max}} = 32.5^\circ$
15310 measured reflections	$h = -10 \rightarrow 10$
	$k = -29 \rightarrow 29$
	$l = -16 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0131P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.052$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.076$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.79$	$\Delta\rho_{\text{max}} = 0.27$ e Å $^{-3}$
5017 reflections	$\Delta\rho_{\text{min}} = -0.17$ e Å $^{-3}$
219 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	2061 Friedel pairs
	Flack parameter: 0.00 (7)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9–H9 \cdots O11 ⁱ	0.93	2.55	3.357 (4)	145
C4–H4 \cdots O31	0.93	2.40	2.730 (4)	101
C9–H9 \cdots Cl7	0.93	2.71	3.071 (3)	104
C21–H21A \cdots O32	0.97	2.30	2.927 (4)	122

Symmetry code: (i) $-x + 1, -y, z - \frac{1}{2}$.

All H atoms were located in difference maps and then treated as riding atoms, with C–H distances of 0.95 (aromatic) or 0.96 Å (methyl) and $U_{\text{iso}}(\text{H})$ values of $1.2U_{\text{eq}}(\text{C})$ (aromatic) or $1.5U_{\text{eq}}(\text{C})$ (methyl). PLATON (Spek, 2003) was used for the hydrogen-bonding analysis.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: OSCAIL (McArdle, 2003) and SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: OSCAIL and SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: CIFTAB (Sheldrick, 1997).

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